APS Applications for Understanding and Modeling the Performance of Reactor Materials under Tensile Loading Conditions

Dr. Xiao Pan, Dr. Jon Almer, Dr. Meimei Li & Dr. James F. Stubbins

ATR User Week
1-5 June 2009

Department of Nuclear, Plasma and Radiological Engineering
University of Illinois at Urbana-Champaign

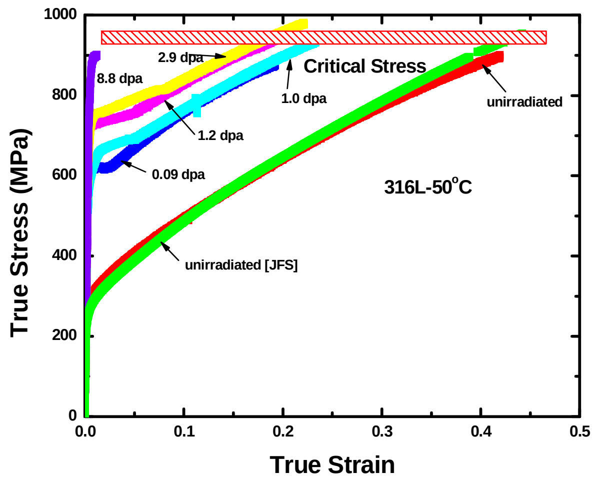
Argonne
NATIONAL
LABORATORY

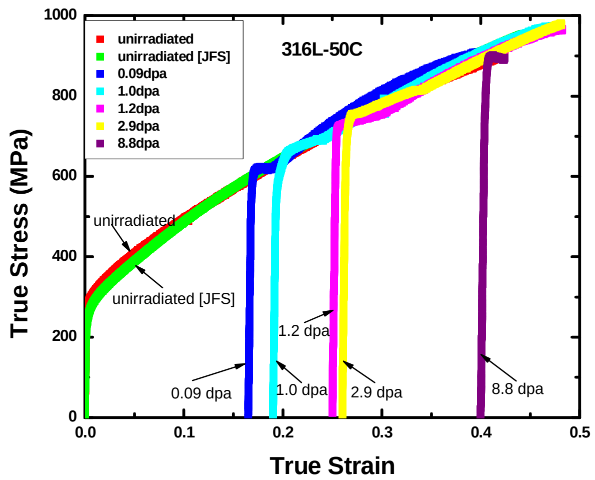
The Problem

- Irradiation-induced embrittlement, or flow localization, is a major concern for advanced nuclear systems
- Tensile behavior is complex with several distinct regions:
  - Elastic, Plastic, Necking and Fracture
- Past experimental approaches cannot examine the controlling processes in each region
- Irradiation has a major influence on the transition between elastic and plastic response
- *However*, irradiation seems to have little effect on plastic strain hardening, plastic instability (necking) and fracture stress
- The plastic and failure response seem to be controlled by a ***critical stress***

The Problem

- **Critical stress** is associated with void nucleation
- Void nucleation is a complex process, depending on interfacial strength, particle size, particle volume fraction, stress state, and matrix strength, etc.
- Several stress criteria have been developed to characterize void nucleation: dislocation model, continuum calculation, and fracture analysis.
- Models have limitations
  - Derived from post-deformation (static) microstructural characterization
  - Contains no information about the dynamic processes during failure





Motivation, Challenge and Reward

Motivation:

- Understand the microstructural features which control tensile deformation – with and without irradiation exposure

Challenge:

- Measure deformation processes at the microstructural level *during* loading

Reward:

- Ability to model tensile behavior:
  - Complete response: elastic, plastic, necking and fracture
  - Control microstructure for optimal tensile performance
  - Relate tensile response to fracture toughness and other key mechanical properties

Objectives and Approaches

Objectives:

- Understanding the controlling mechanism of the critical stress
  - Correlation between critical stress and interfacial strength for void nucleation
- Understanding the characteristics of the critical stress
  - Effects of particle size, particle distribution and orientation, and particle volume fraction.
- Understanding the temperature dependence of the critical stress.

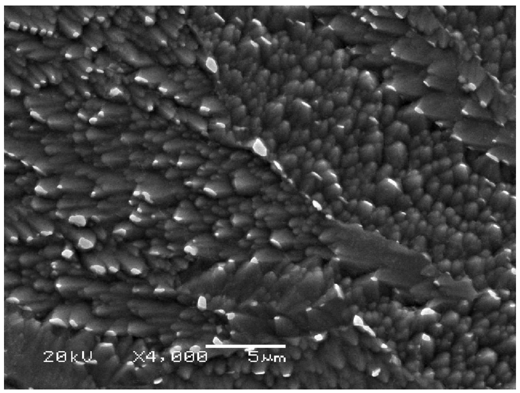
Approaches:

- Tensile tests at 20, 100, 200, 300 and 400°C
- *In situ* tensile tests with x-ray diffraction (XRD) and small angle x-ray scattering (SAXS)
- Ultra-small angle x-ray scattering (USAXS)
- Microstructural analysis by scanning electron microscopy (SEM)

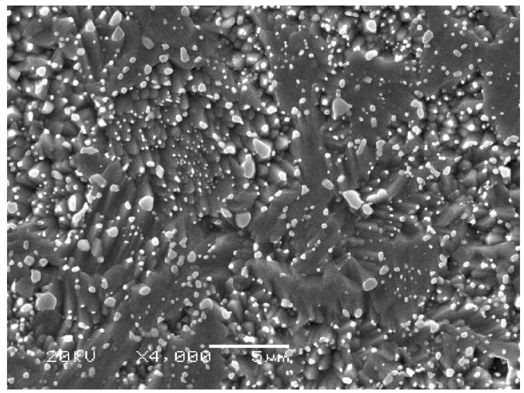
Materials: 9-12Cr Ferritic Model Alloys

Heat Treatment:

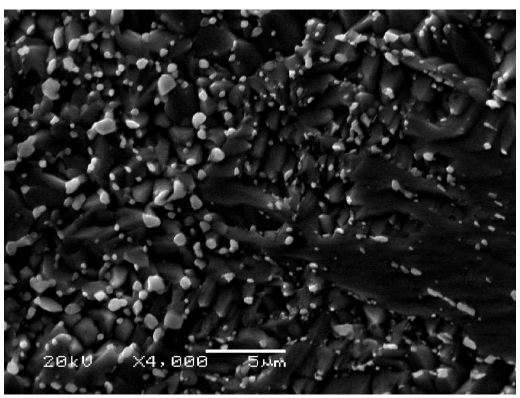
- Austenitisized at 980˚C/0.5 h &air cooled
- Tempered at 720˚C for 4, 8, 16 days



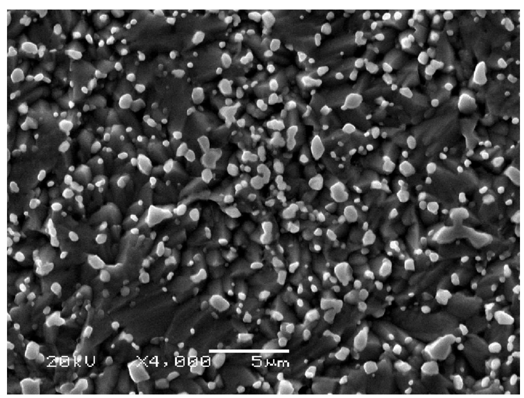
Fe-9%Cr-0.1%C



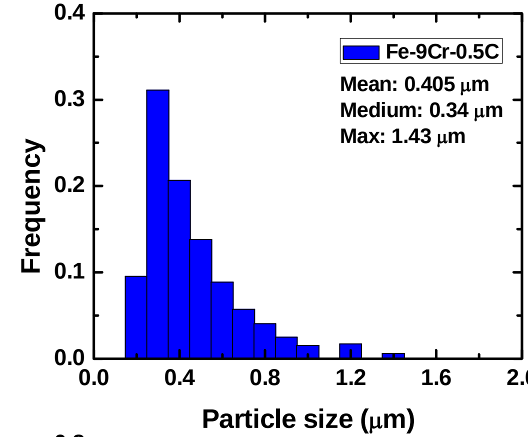
Fe-9%Cr-0.5%C

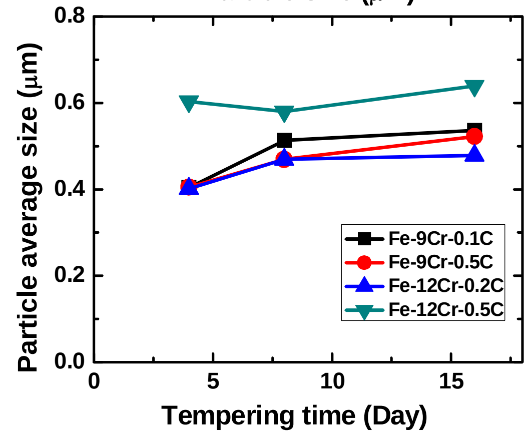


Fe-12%Cr-0.2%C



Fe-12%Cr-0.5%C

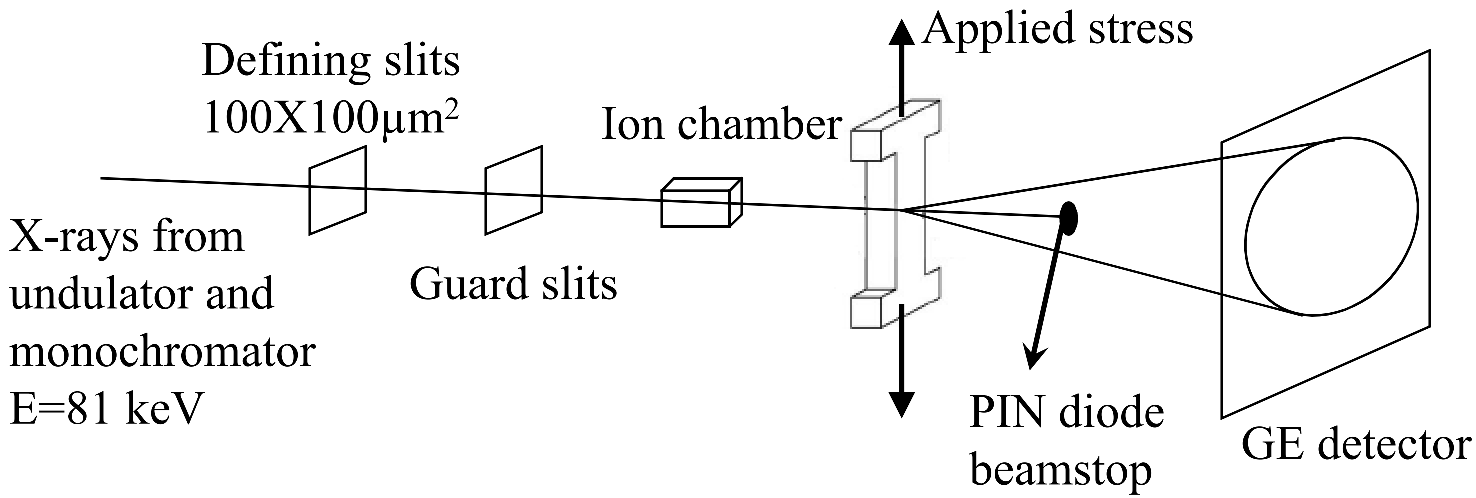




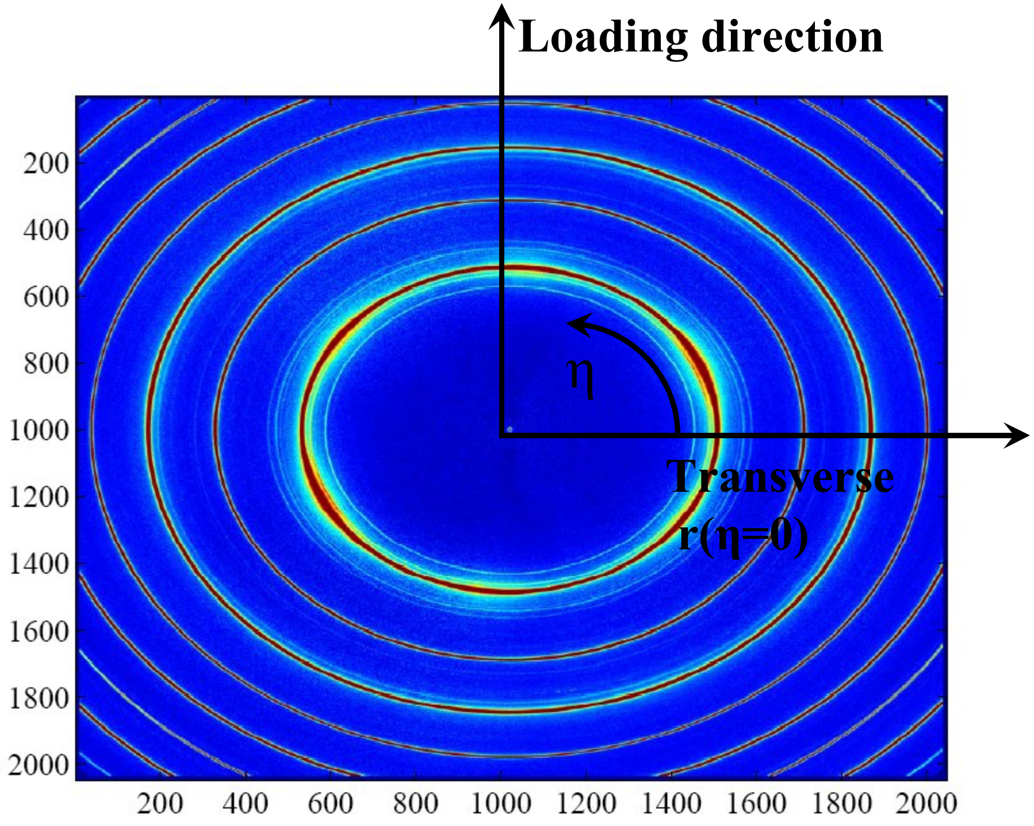
- ♦ Particle size increases with tempering time
- ♦ Particle volume fraction increases with C%

In situ x-ray Measurements

- APS 1-ID beamline, 81 keV (λ= 0.015 nm) x-ray beam with a square 100×100 μm^2.
- *In situ* tensile test by two steps: continuous and interrupted test.



X-ray Diffraction Analysis



Lattice Strain can be expressed by the radius change of Debye–Scherrer diffraction rings

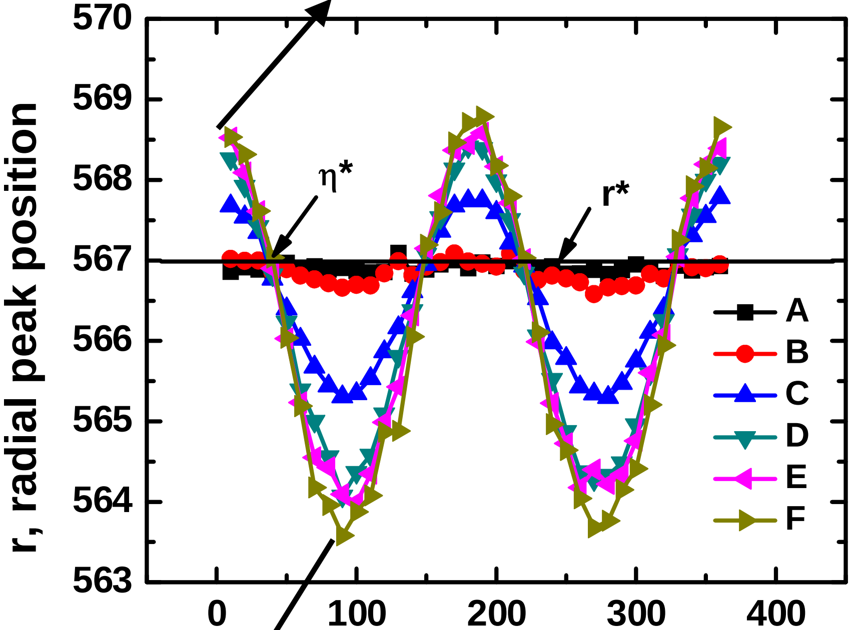
$$\varepsilon_\eta = \frac{r^* - r_\eta}{r^*}$$

$$\varepsilon_{22} = \varepsilon_{90^0}$$

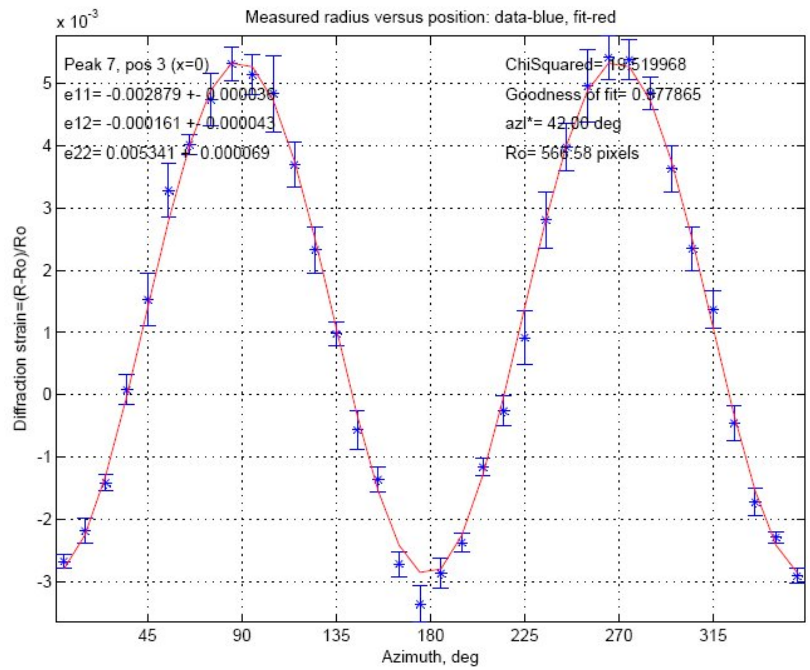
$$\varepsilon_{11} = \varepsilon_{0^0}$$

X-ray Diffraction Analysis

Transverse direction



Loading direction η (deg)



Radial position vs. Azimuthal degree

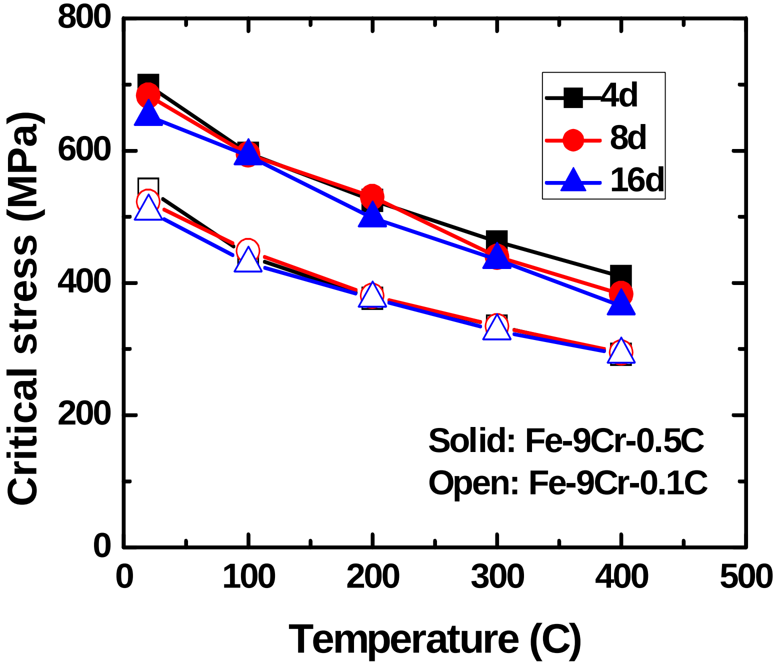
$$\varepsilon_\eta = \frac{r^* - r_\eta}{r^*}$$

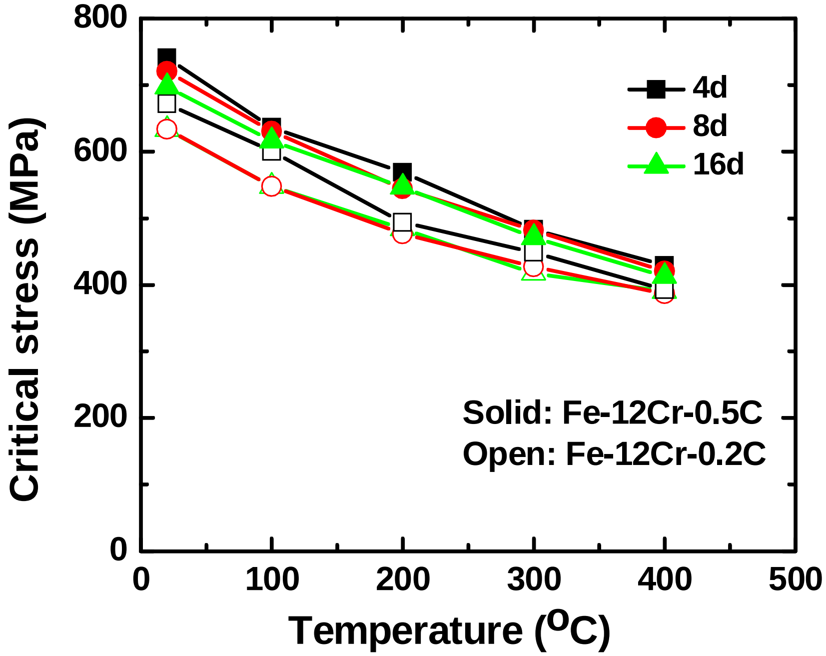
$$\varepsilon_{22} = \varepsilon_{90^0}$$

$$\varepsilon_{11} = \varepsilon_{0^0}$$

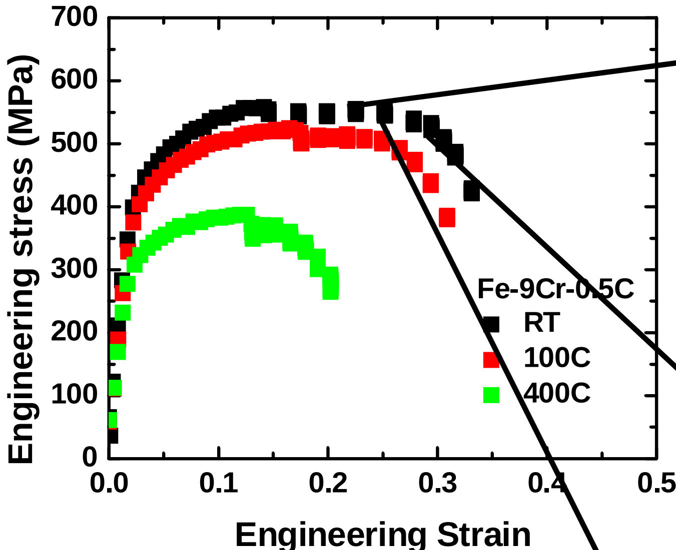
Experimental Results - Critical Stress

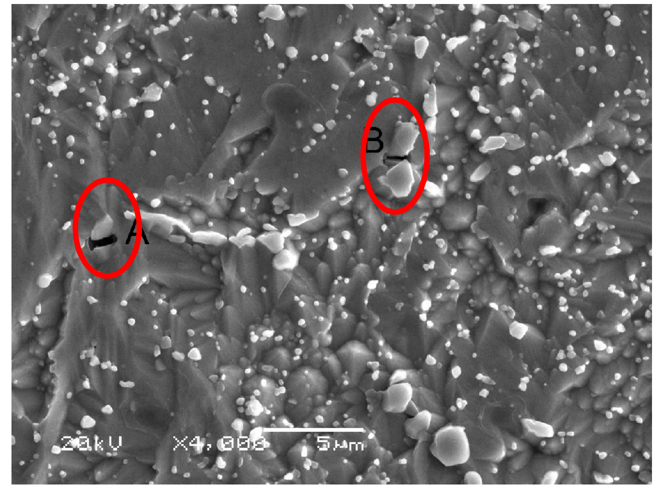
- Critical stress decreases with increasing temperature
- Critical stress decreases with increasing particle size
- Critical stress increases with increasing carbon concentration, i.e. particle volume fraction



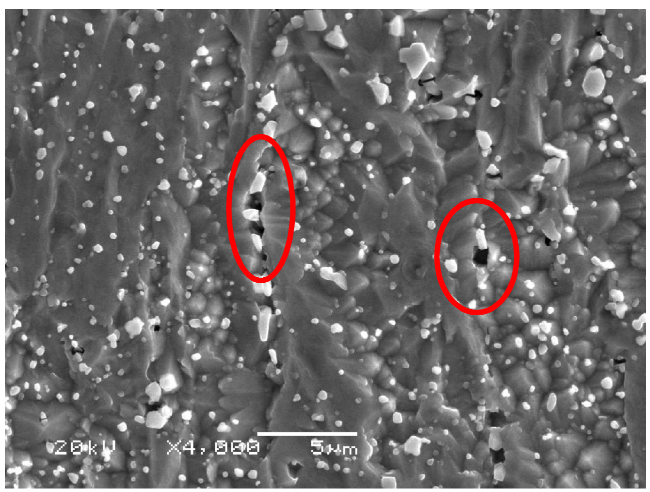


Void Evolution by SEM

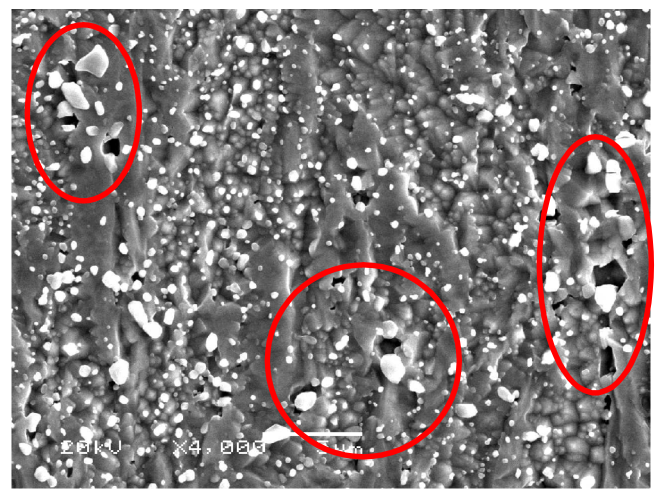




UTS, ε=0.225



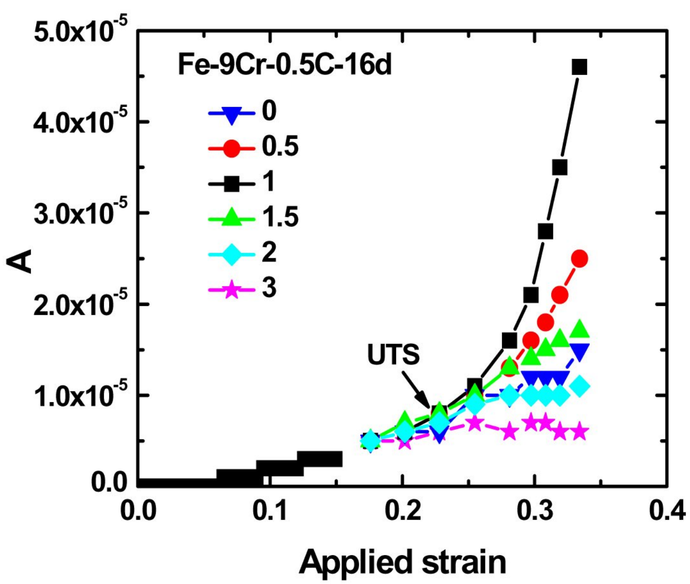
UTS, ε=0.35

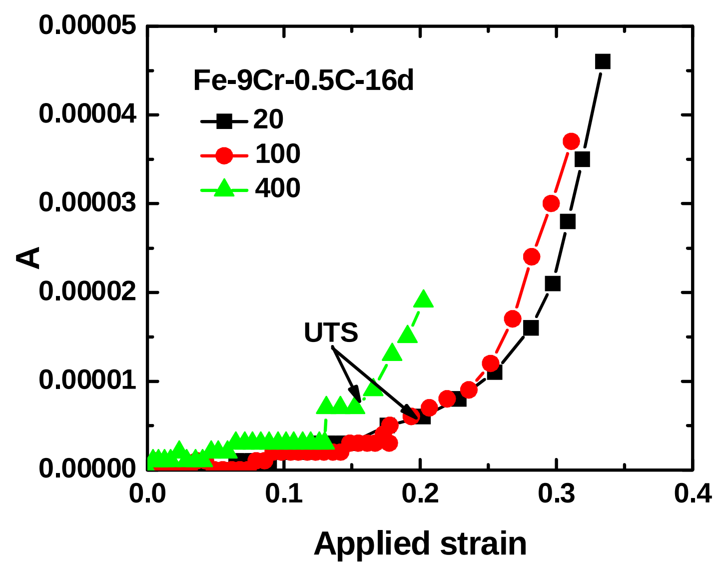


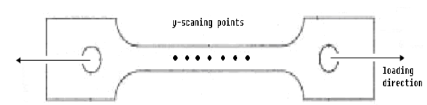
UTS, ε=0.55

Void Evolution by *in situ* SAXS and XRD

- Void density is low and uniform before UTS.
- After passing UTS, void density is location dependent.
- Void density decreases with increasing temperature.



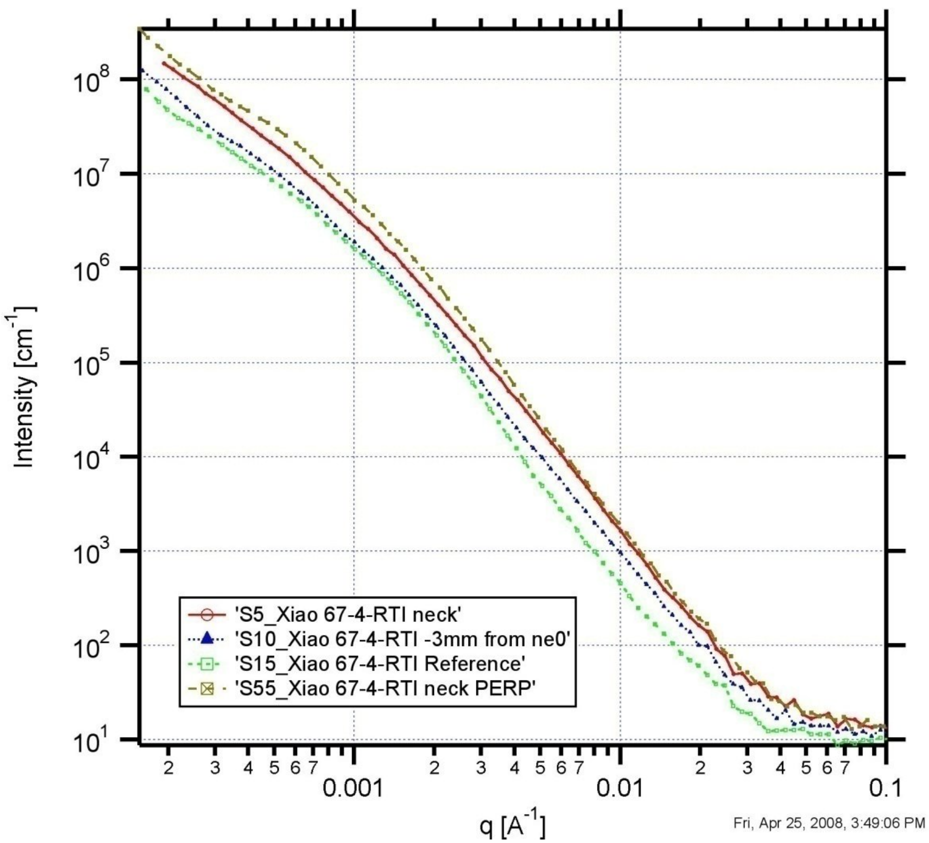




$$I = Aq^{-4} + B$$

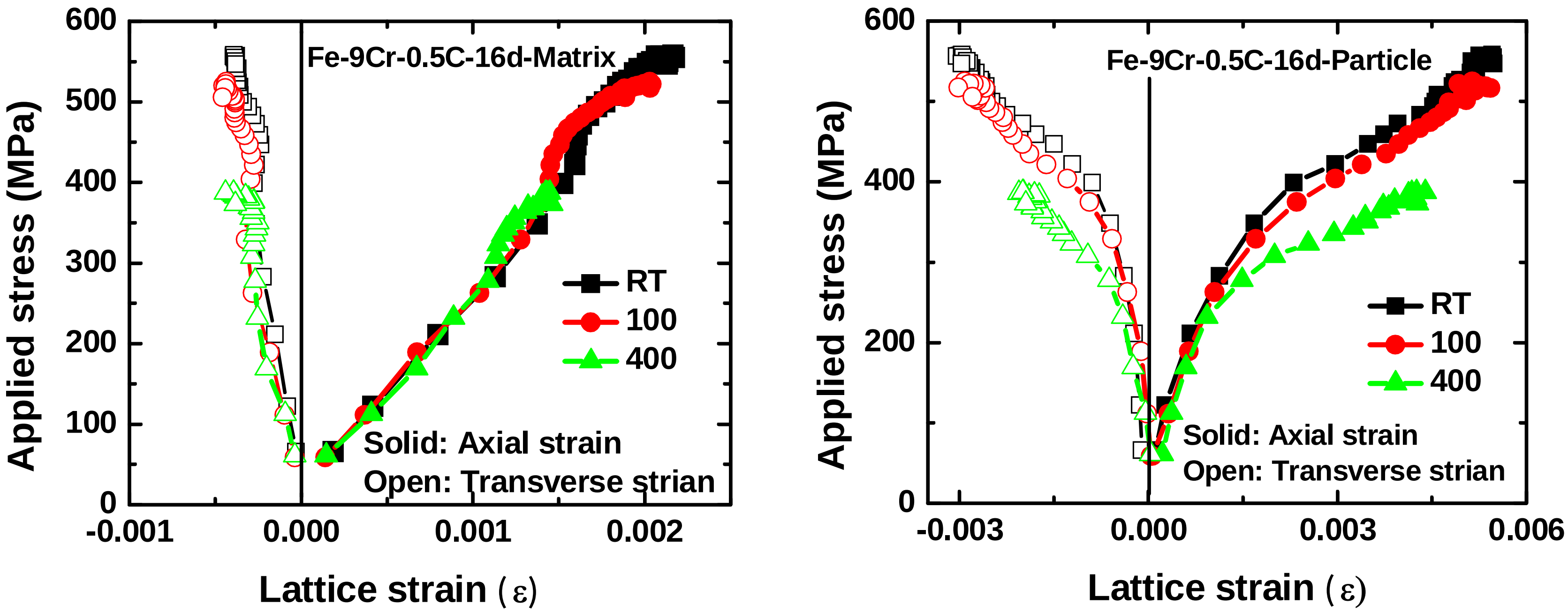
Void Evolution by USAXS

USAXS measurements cover the Q-value in the range from 0.0003 to 0.04 $Å^{-1}$ (feature size from 15 to 200 nm.)



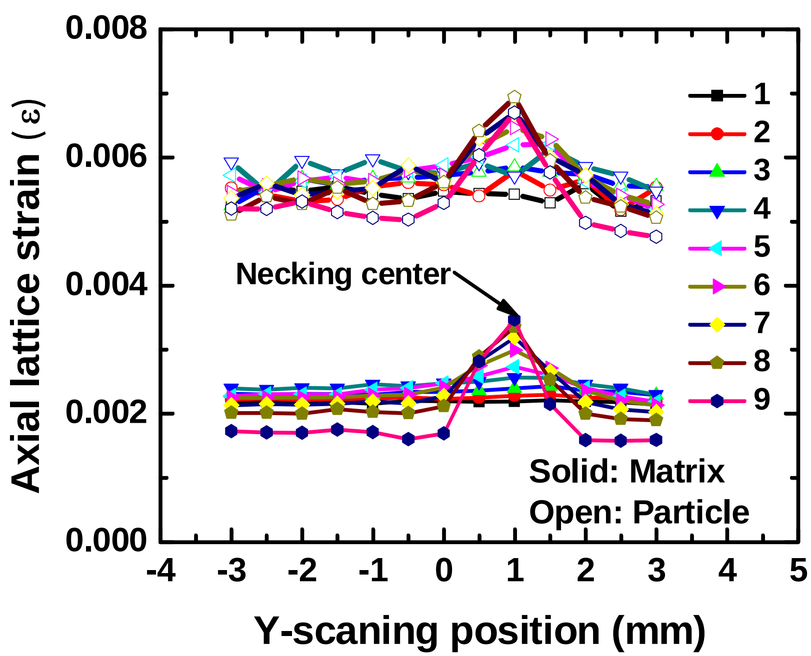
Lattice Strain Evolution

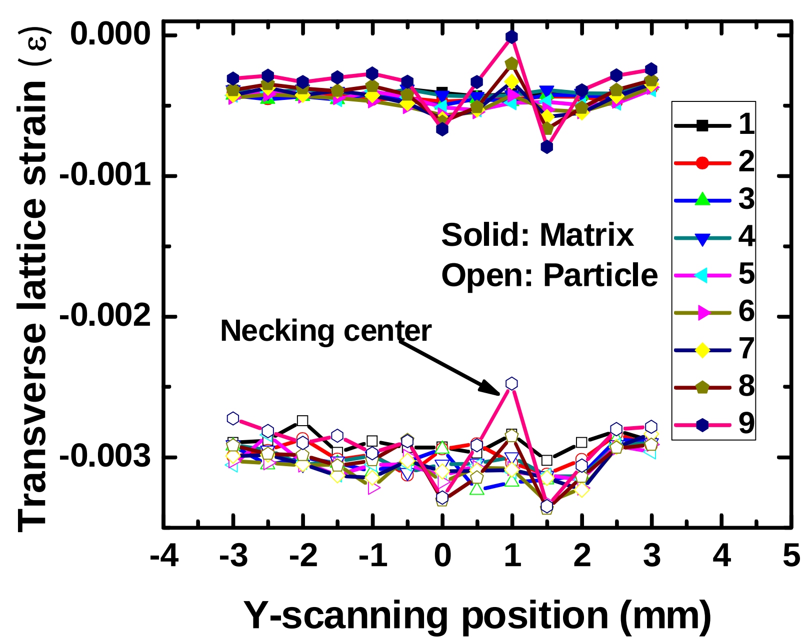
- Temperature has different effect on particle and matrix.
- Load partition occurs earlier with increasing temperature.
- Near-zero mismatch between particle and matrix at elastic region.
- Load transfer starts at the beginning of yield point.



Lattice Strain Evolution

- Axial lattice strain increases quickly near necking point due to strain localization, and is flat far away from necking region.
- Decrease of absolute value of transverse lattice strain at necking point is due to hydrostatic tension stress.





Critical Interfacial Strength

Comparison of critical interfacial strength determined by lattice strain measurements and the dislocation model

Interfacial strength (MPa)	Fe-9%Cr-0.5%C-4d	Fe-9%Cr-0.5%C-8d	Fe-9%Cr-0.5%C-16d
This work	1162	1120	1065
Dislocation model (r = average size)	1490 (0.2µm)	1426 (0.23µm)	1344 (0.26µm)
Dislocation model (r = large size)	1089 (0.4µm)	1044 (0.4µm)	921 (0.5µm)

X-ray measurement:

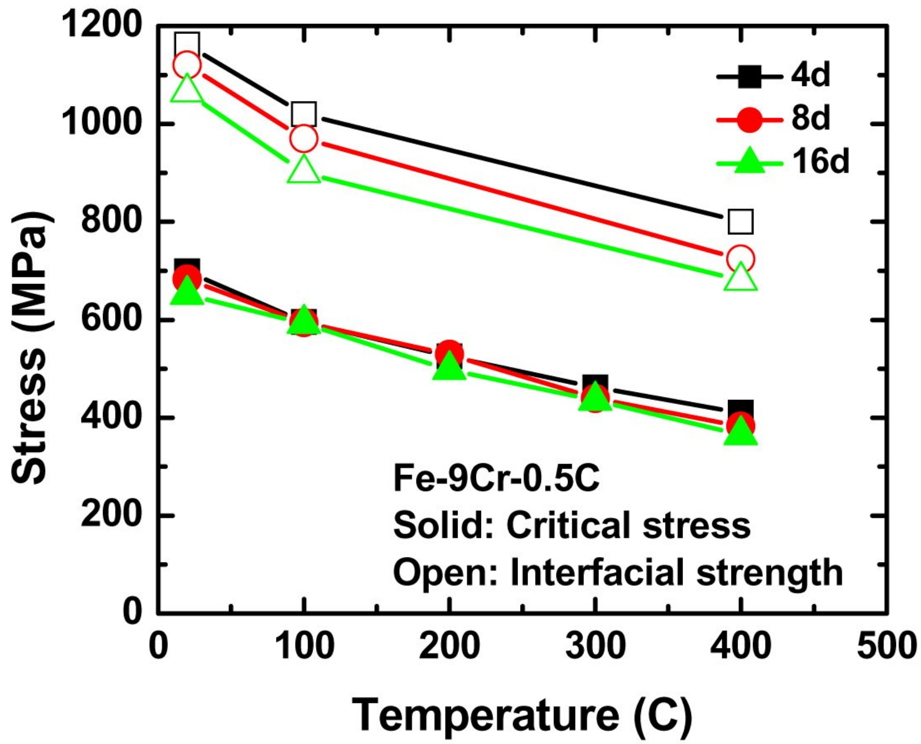
$$\sigma_{22} = \frac{E}{1+\nu}\varepsilon_{22} + \frac{\nu E}{(1+\nu)(1-2\nu)}(\varepsilon_{22}+\varepsilon_{11}+\varepsilon_{33})$$

$$\sigma_{11} = \sigma_{33} = \frac{E}{1+\nu}\varepsilon_{11} + \frac{\nu E}{(1+\nu)(1-2\nu)}(\varepsilon_{11}+\varepsilon_{22}+\varepsilon_{33})$$

Dislocation model:

$$\sigma_{interfacial} = 4.48\times10^{-3} r^{-0.7}(\varepsilon_n)^{0.3} + 1.75\times10^{-2}\mu(\varepsilon_n / r)^{0.5} + \sigma_T/(1-f_v)$$

Critical Interfacial Strength

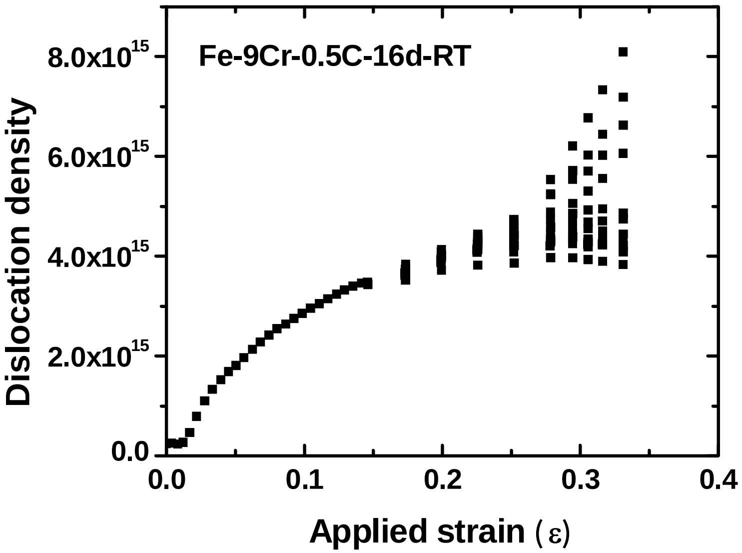


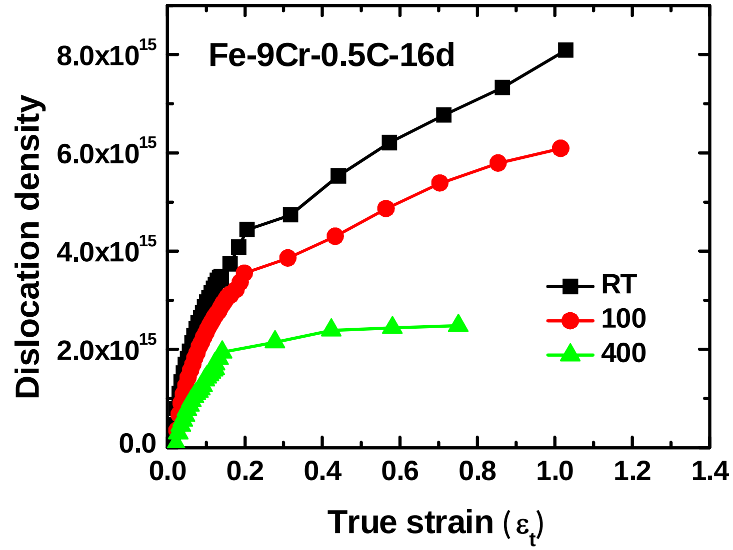
- Critical stress is linearly correlated with critical interfacial strength

$$\sigma_{interfacial} \approx (1.5 \sim 1.8)\sigma_{critical}$$

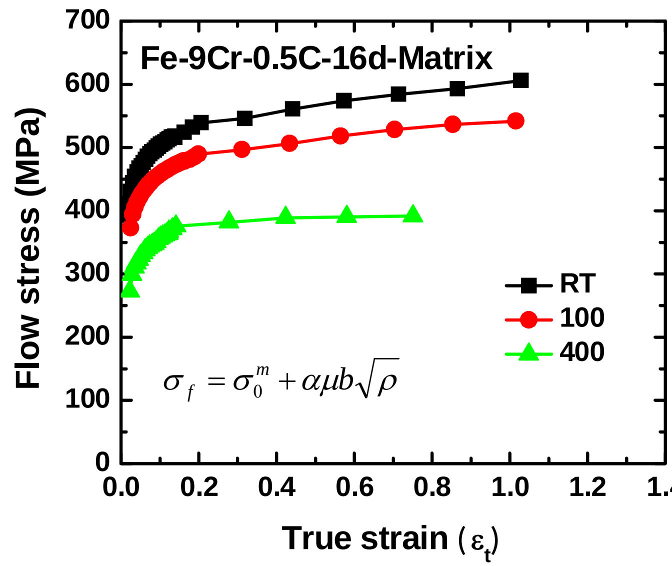
- Both critical stress and interfacial strength have strong temperature dependence

Dislocation Density Analysis





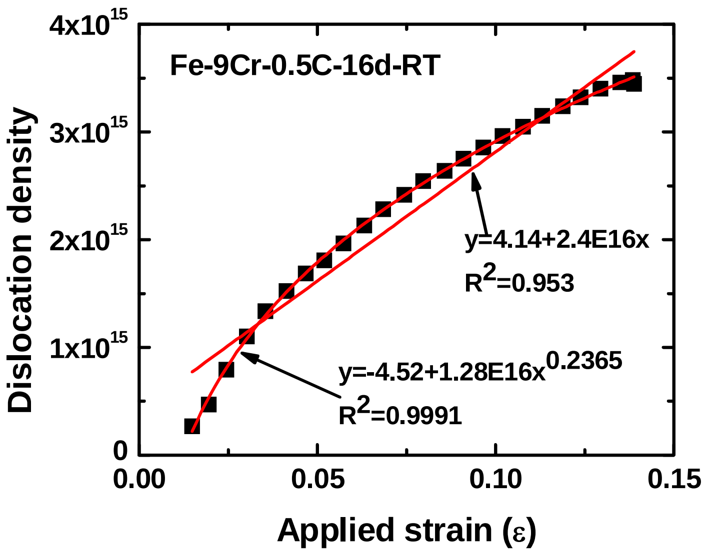
- Dislocation density determined by x-ray peak profile analysis
- Dislocation density continuously increase with strain
- There is a scattering behavior after passing UTS



Micro-Structural Model (MSM)

Micro-structural model

$$\sigma^p = (\sigma_t - (1 - f_p)(\sigma_0 + \alpha\mu b\sqrt{M\varepsilon^N})) / f_p$$

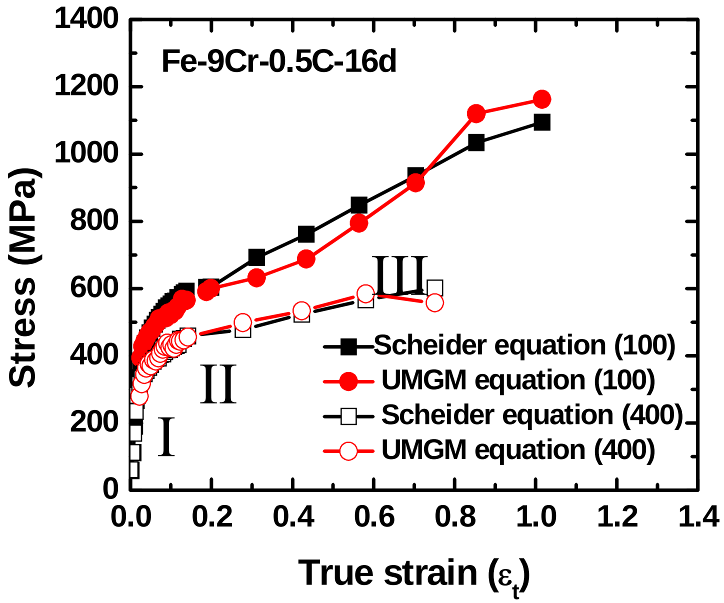


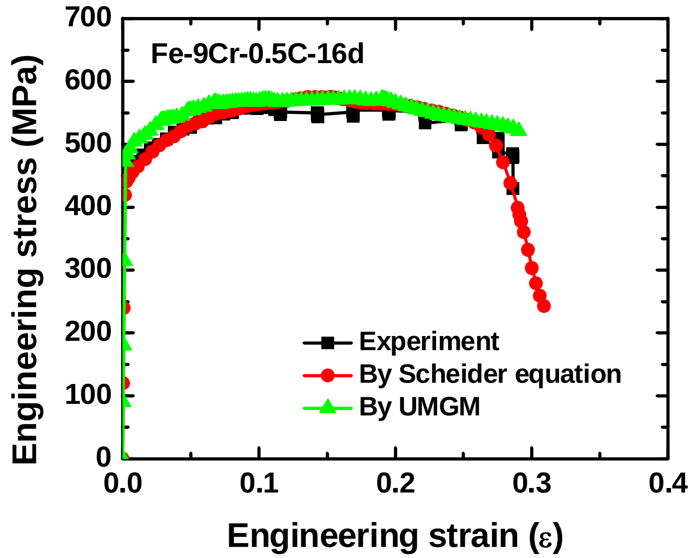
Material	X-ray measurement	MSM model	Dislocation model
Fe-9%Cr-0.5%C 4d-RT	1162	1204	1490 1089
Fe-9%Cr-0.5%C 8d-RT	1120	1193	1426 1044
Fe-9%Cr-0.5%C 16d-RT	1065	1034	1344 1048
Fe-9%Cr-0.5%C 16d-100	900	945	1390 1085
Fe-9%Cr-0.5%C 16d-400	680	690	1193 930
Fe-9%Cr-0.1%C 4d-RT	1350	1505	1800 1250
Fe-12%Cr-0.1%C-4d-RT	1342	1432	1586 1145
Fe-12%Cr-0.5%C-4d-RT	1390	1378	1281 1087

MSM calculations match x-ray measurements for all tested cases and provide better prediction than dislocation model.

Universal Microstructural Geometry-corrected Model (UMGM)

UMGM Model: $$\sigma_t = \sigma^m (1 - e^{1.5\varepsilon_t} f_p) + e^{1.5\varepsilon_t} \sigma^p f_p$$





♦ UMGM calculations are consistent with experimental data, and evaluated by FEM analysis.

♦ Three stages of deformation: elastic, plastic, and post-necking region.

$$I : \sigma_t = E\varepsilon_t \qquad II : \sigma_t = K\varepsilon_t^m \qquad III : \sigma_t = C\varepsilon_t$$

Conclusions

- UTS is a critical and starting point for void nucleation. Void nucleation is controlled by the critical interfacial strength.
- Critical stress is linearly correlated with critical interfacial strength.
- Critical interfacial strength has strong temperature dependence.
- Particle characters have significant effects on critical interfacial strength.
  - Decreases with increasing particle size,
  - Dependent on particle morphology.
  - Independent of particle volume fraction
- Micro-Structural Model (MSM) can predict critical interfacial strength using macro tensile testing parameters. MSM model is consistent with experimental measurements and provides better prediction than dislocation models.
- Universal Micro-structural Geometry-corrected Model (UMGM) offers new method to calculate true strain-true stress curve up to fracture. Grounded with experimental data, the UMGM provides empirical validation for existing methods.

Future Work

- ATR Irradiation Program – in preparation
- *In situ* void growth and coalescence at APS
- Irradiation-induced flow localization
- Notch effect and strain mapping
- More precise experimental design